



Parameter and Sample Type

Calcium in Fruit Juice

Introduction

This method provides rapid, uncomplicated determinations for the free calcium ion in fruit juice. The direct measurement of ions using an ion selective electrode is a well established and frequently used standard method of analysis. This analysis is used as a quality control step and to fulfill labeling requirements. This procedure eliminates interferences associated with presence of color and solids.

Result Statistics

| # Trials | Average | %CV |
|----------|---------|------|
| 5 | 41.6ppm | 2.2% |

Recommended Equipment

4-Star Benchtop pH/ISE meter (Orion 1115000); ionplus[®] calcium electrode (Orion 9720BNWP); Benchtop stirrer (Orion 096019) Optional: ATC probe (Orion 927007MD)

Required Solutions

Calcium Standard, 0.1M as CaCl₂ (Orion 922006); Calcium ISA Solution (Orion 932011); Reference Filling Solution, Optimum Results A (Orion 900061); Deionized (DI) water.

Solutions Preparation

Prepare a 40ppm calcium standard by pipetting 1mL of 0.1M (4000ppm) calcium standard in a 100mL volumetric flask, add 2mL of calcium ISA and dilute to the mark with DI water. Mix well. Prepare a 4ppm calcium standard by pipetting 10mL of the 40ppm calcium standard in a 100mL volumetric flask, add 2mL of calcium ISA and dilute to the mark with DI water. Mix well. Use 30mL of each of these standards for calibration.

Meter Setup

Connect the electrode, stirrer and ATC probe to the Star Meter. Set measurement mode to ISE. In Setup mode of Star Meter, set resolution to 3, set the measurement range to high, select ppm as the unit and read type to continuous. If all steps were followed correctly the meter display will show three digits in the top line and "ISE: ppm" to the right of the top line. The temperature will also be displayed in the top left of the screen.

Electrode Setup

See the electrode manual for preparation of the electrode.

Electrode Performance Check

Check slope at least daily according to the electrode manual. Drift may be checked by comparing a 1 minute to a 2 minute reading. Results should agree with desired criteria. See troubleshooting section of manual if slope or drift problems.

Electrode Storage, Soaking, and Rinsing

See electrode manual for storage 1) between measurements, 2) overnight, and 3) for long periods of time. Between measurements, rinse the electrode with DI water and dry outer sleeve of electrode before measuring the next sample. Do not wipe or rub the sensing element of the electrode, shake the electrode to dry the sensing element.

Sample Preservation

None required.

Sample Preparation

Pipette 50mL of fruit juice into a 200mL volumetric flask. Dilute to the mark with distilled water and mix well.

Calibration

Perform a two point calibration using the 4ppm and 40ppm calcium standards. The electrode slope will be displayed and should be between 25 and 30mV/decade. Read a fresh portion of standards to verify calibration. If readings are not acceptable see troubleshooting section of manual.

Analysis

Rinse electrode, ATC probe and stirrer with DI water and shake to probe to dry. Pour a 50mL portion of the sample, and add 1mL ISA to the sample aliquot. Place probes in sample, turn on stirrer and measure. The concentration of the sample will be displayed. When a stable reading is achieved, the "ISE:ppm" icon will stop flashing. Multiply the meter reading by the dilution factor (in this case 4).

Quality Control (QC)

Recommended QC procedures include: calibration and calibration verification, sample duplicates, slope, matrix spikes, lab control samples and MDL.



| Fruit Juice | Direct Readings Calcium (ppm) | Final Results Calcium (ppm) |
|--------------------|-------------------------------|-----------------------------|
| Sample 1 | 10.1 | 40.4 |
| Sample 2 | 10.3 | 41.2 |
| Sample 3 | 10.5 | 42.0 |
| Sample 4 | 10.7 | 42.8 |
| Sample 5 | 10.4 | 41.6 |
| | | |
| Mean | 10.4 | 41.6 |
| Standard Deviation | 0.2 | 0.9 |
| %CV | 2.2 | 2.2 |

Note: the corrected readings of the ppm free calcium in fruit juice were obtained by multiplying the direct readings by the dilution ratio of 4 due to the sample preparation. The final average result was 41.6ppm free calcium in the fruit juice.